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Original Article

Rheology of diluted and semi-diluted partially hydrolyzed polyacrylamide solutions under shear: Experimental studies

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ABSTRACT

Rheological properties of hydrolyzed polyacrylamide (HPAM) solutions were measured in oscillatory and flow shear. In oscillatory shear the storage and loss moduli increased as the concentrations of the solutions increased, but they decreased as salinity increased. The relaxation spectra were plotted using the Kontogiorgos method. As shown in the relaxation spectra, the number of density of segmental units increased at first and then the peaks shifted and split into several as the concentration increased. With salinity increasing, the number of motion units increase and the peaks of segmental units became broader. In flow shear tests, the curves of viscosity versus shear rate were fitted by a power law equation. The result showed that the flow behavior index decreased with increasing concentration, while it generally increased with increasing of salinity. Furthermore, the first normal stress difference increased as the concentration was increased, while it decreased as the salinity was increased. The first normal stress differences measured by the rheometer were compared with the first normal stress differences calculated by the Laun equation and the results showed that Laun equation deviates the experimental results.

1. Introduction

Polymers, for soft natures, has been widely applied in the petroleum industry [1]; for example crude oil pour-point depressant [2,3], adhesives for petroleum pipeline coating [4–6], oil well cement modifier [7], polymer flooding agents [1,8] and so on. This is because of the long chain nature of polymers, displaying both strong viscosity and elasticity (called viscoelasticity) [9]. And the multiple molecular modes of polymers involve in dynamics from low to high temperatures; meanwhile coupling and decoupling environments make rheology of polymers become complex [10–14].

Nowadays, HPAM, as an effective flooding agent, has been widely used in tertiary oil recovery. In the Chinese petroleum industry, the use of the flooding agent HPAM is at the industrial application and dissemination stage, especially in enhanced oil recovery of the late exploitation stage [1,15]. Usually, HPAM is prepared by hydrolyzing polyacrylamide or copolymerization of acrylamide and acrylate. The hydrolysis degree of HPAM used in enhanced oil recovery is often about 25%–35%. On one side, electrostatic interaction from carboxyl groups in HPAM molecular chains would make molecular coils become extended [16,17]. A higher hydrolysis degree can cause an increasing solution viscosity. On the other side, a higher hydrolysis degree also can increase the sensitivity of the HPAM to salt [18], which can decrease the viscosity of the HPAM solution. Thus, the rheological properties of HPAM solutions are complex [19].

In contrast to other polymer displacement agents, HPAM displacement agents have a high molecular weight and good

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performance in thickening; even diluted HPAM solutions have a high viscosity. Some scholars [20] think that the improvement in oil recovery ratio is mainly caused by the high viscosity. The high viscosity of the HPAM solutions increase the recovery ratio through improving the sweep coefficient, mobility ratio and water-injection profile. In addition, Wang et al. [21–23] indicated that the elasticity of HPAM solutions also have an apparent influences on oil flooding. Furthermore, Liu et al. used numerical

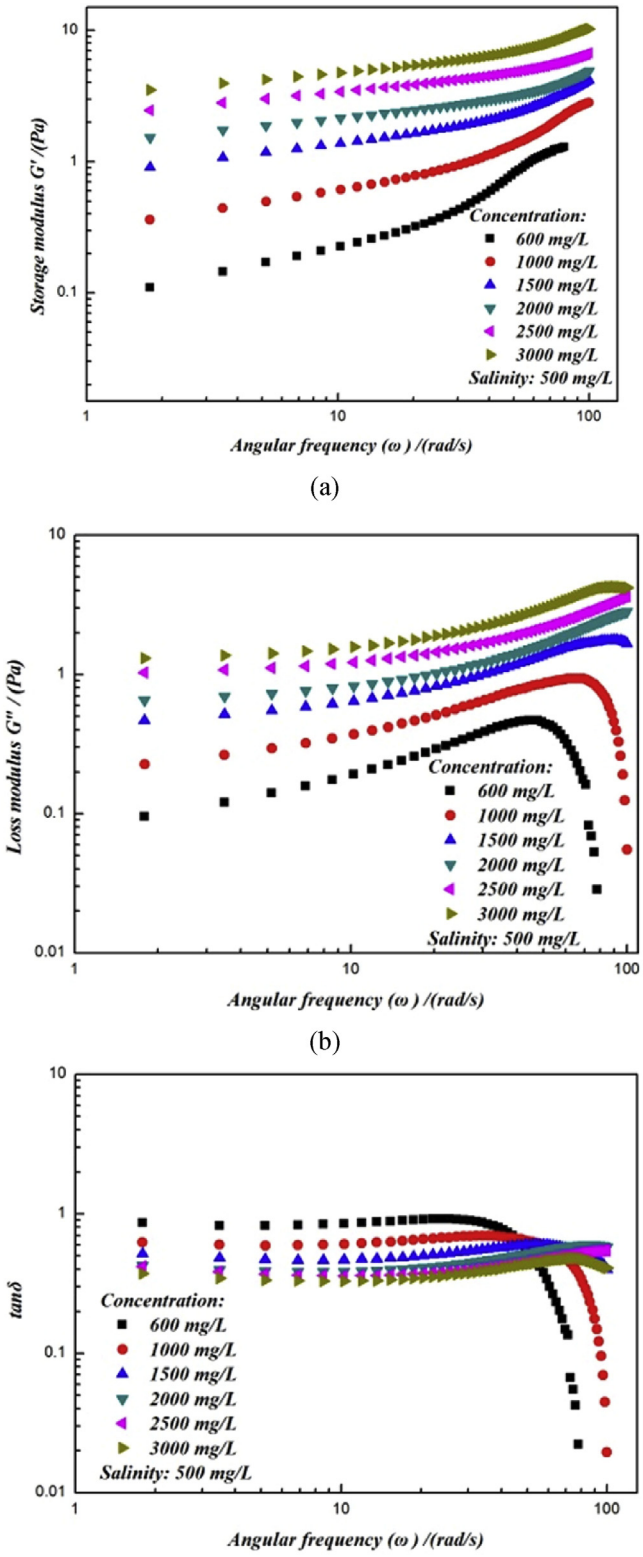

Fig. 1. Variation of storage modulus G′ (a), loss modulus G″ (b) and tanδ (c) of HPAM solutions with different concentrations as a function of angular frequency.

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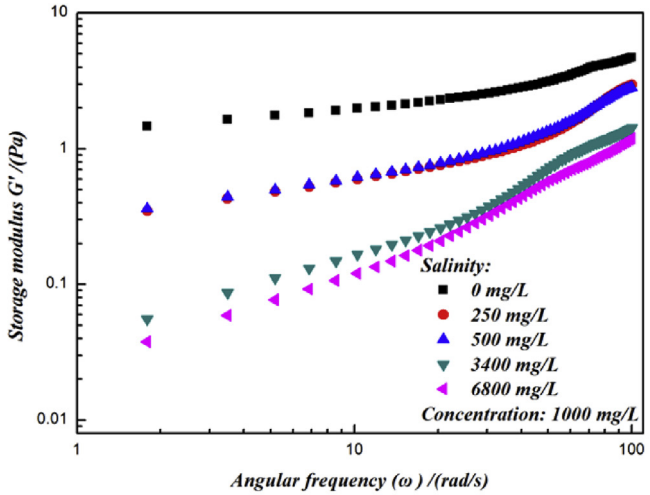

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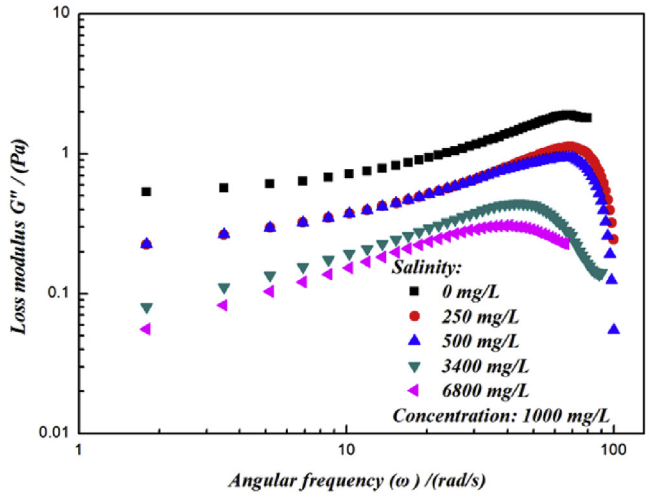

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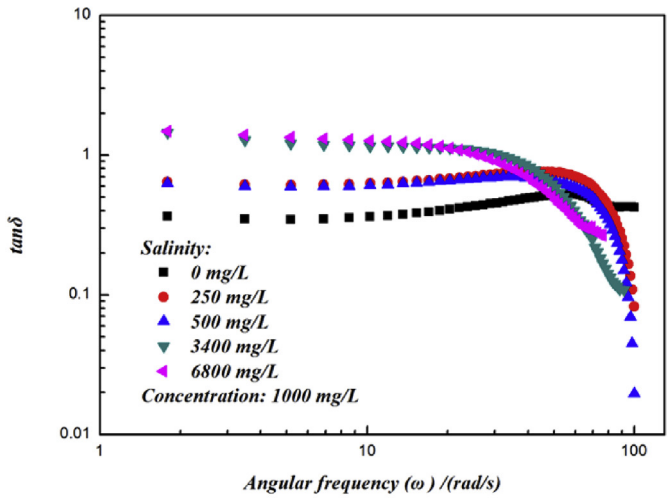

(c)

Fig. 2. Variation of storage modulus G′ (a), loss modulus G″ (b) and tanδ (c) of HPAM solutions with different salinities as a function of angular frequency.

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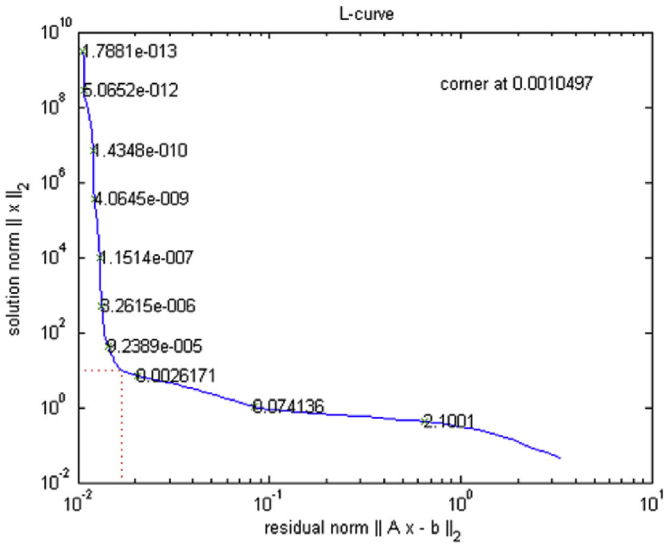

Fig. 3. Curve of solution norm versus residual norm (L-curve) of HPAM solution with concentration of 1000 mg/L and salinity of 6800 mg/L.

simulation and indicated that HPAM polymer solutions have a wider mobile area in porous medium than Newtonian fluids. They also showed that first normal stress difference is an important element in oil flooding. HPAM polymer solutions with higher first normal stress difference would have higher interaction with the oil face in porous medium [24]. Thus, both elasticity and viscosity have apparent contributions to oil flooding; elasticity and viscosity also have close relationships with the oscillatory shear and flow shear behaviors.

Some scholars have found that the relaxation time has a close relationship with the elasticity of polymer solutions. Based on the parallel pore throats model, Zhang et al. indicated that the effective relaxation time of a polymer increases as the radii of the pore throats increase [25]. In addition, Zhang et al. proposed that the relaxation time has an important effect on the oil recovery ratio through a simulated 3D viscoelastic polymer flooding model [26]. Unfortunately, the related works studying the rheological properties of HPAM solutions usually have obtained the relaxation time through the oversimplified Maxwell model, but the distribution of relaxations times was not considered [27]. However, Ferry indicated that the distribution of relaxation times has an important influence on rheological properties of HPAM solutions [28]. Many viscoelastic studies have shown that different relaxation processes have different effects on the mobilities of polymers [29]. Furthermore, the first normal stress difference also has a close relationship with the elasticity of polymer solutions. Generally speaking, the greater first normal stress difference of polymers means the polymers have larger elasticity. However, calculations of the first normal stress difference have been obtained through a semi-empirical equation [30]. But this equation herein which estimated the first normal stress difference through storage modulus and loss modulus are obtained from studying polymer melts [31,32]. However, with the development of rheometers, the first normal stress differences of polymer solutions with low concentration can be measured by some rheometers by eliminating the inertial system and using strain controlled modes [33].

In the present paper the oscillatory shear and flow shear behaviors of HPAM dilute and semi-dilute solutions are described. The relaxation time spectra were drawn in terms of the storage modulus. In a related work from our laboratory, an ill-posed problem usually happened in calculating the relaxation time spectra [34]. Herein, Kontogiorgos's method was used to avoid the ill-posed problem. Furthermore, the flow characterizations were described by a power law [35]. Finally, the first normal stress difference is discussed.

2. Experimental method

The HPAM (industry grade) was supplied by the Geological Research Institute of Shengli Oil Field, P. R. China. The M_w of the HPAM was about 2.30×10^7 g/mol. Sodium chloride (NaCl, analysis pure grade) was purchased from Changzheng Chemical Co. Ltd., P. R. China. The molar mass is 58.44 g/mol. The high purity water was prepared in a Water Purification System (UPZGK, Chengdu Ultrapure Technology Co., Ltd., P. R. China).

Prior to its use the HPAM were dried in a drying oven at 40 °C about 48 h (DHG-9030A, Shanghai Hongdu Electronic Technology Co., Ltd., P. R. China). Solvent (high purity water) and NaCl were subsequently added to the bottle in which the HPAM was weighed after drying. A polytetrafluoroethylene blender was used to stir the solution with a stir rate of 300 r/min. The dissolving temperature was set constantly to 40 °C. After about 72 h, in order to homogenize the solution, the mixture was then transferred into the container cell of the rheometer and other containers.

The rheological properties of HPAM solutions were characterized on an Advanced Rheometric Expansion System (ARES) (G2, TA Instruments Co., USA) using the flat plates for both oscillatory shear method and flow method. At given time intervals frequency sweeps, extending from 100 to 0.1 rad/s in the oscillatory shear method and 100 to 0.1/s in the flow method, were carried out. The testing temperature was set at 25 °C. The relaxation spectra were drawn using the Kontogiorgos method [36,37] through a MATLAB programme (Mathworks, R2009a); the NLC SmoothReg [38] and Hansen package [39] were used to smooth data.

3. Results and discussion

3.1. Oscillatory shear behaviors

In an oscillatory shear test, a polymer solution undergoes oscillatory flow at small amplitude at a chosen frequency. Unlike

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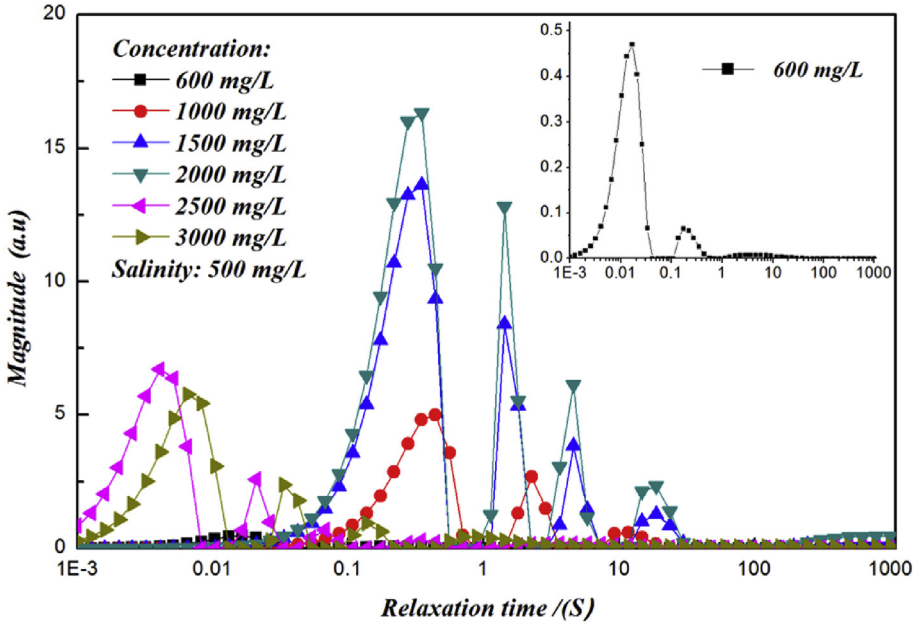

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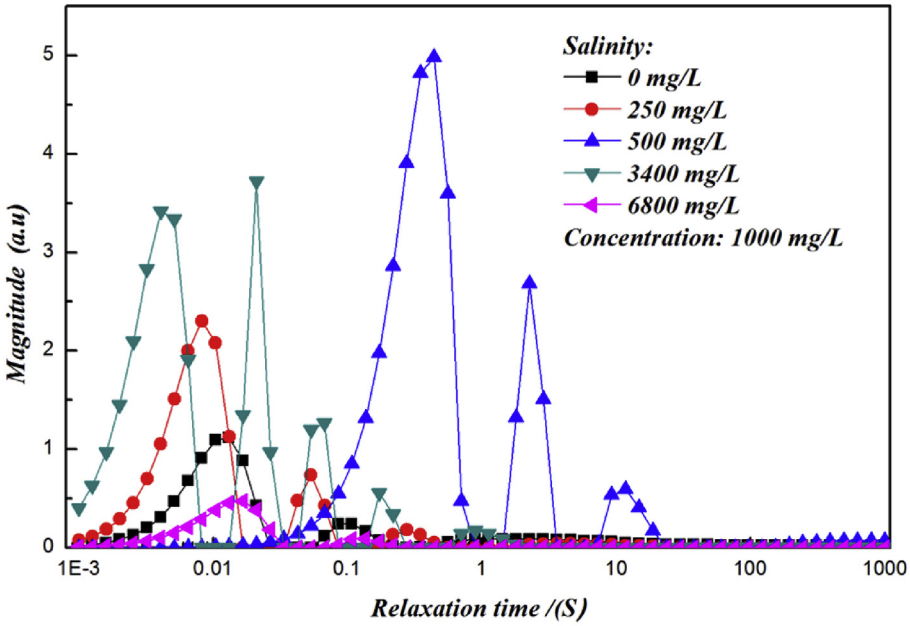

(b)

Fig. 4. Relaxation time spectra of HPAM solutions with different concentrations (a) and salinities (b).

the flow shear test, due to the reciprocating change in the flow direction at the selected frequency, the test results can reflect the dynamic mechanical properties of polymer solutions and yield information about molecular motions. As shown in Figs. 1(a) and 2(a), the storage modulus increased as the angular frequency was increased because the rigidity of polymer solutions increases at high angular frequency. As shown in Figs. 1(b) and 2(b), as the angular frequency increased, the loss modulus increased at first and it then decreased when the angular frequency exceeded a certain value. The turning points in the loss moduli curves are usually thought of as characterization points of local segments motions [40,41]. As shown in Figs. 1(c) and 2(c), the dissipation factor loss tangent (tanδ) remained at a nearly constant value at low angular frequency, while it sharply dropped when the angular frequency increased beyond a certain value. Qualitatively, the drop points may correspond to the freezing of longer units of segments. The drop points shifted to higher angular frequencies as the concentration was increased; however, they shifted to lower angular frequencies as the salinity was increased. The polymer coils would be more crowded as the concentration was increased, so their relaxation times became shorter and the drop points shifted to higher angular frequencies. The difference between 600 mg/L and 1000 mg/L in Fig. 1 is obvious, because the 600 mg/L corresponds to dilute solution but 1000 mg/L corresponds to a transitional situation between semi-dilute solution and dilute solution. Also, the

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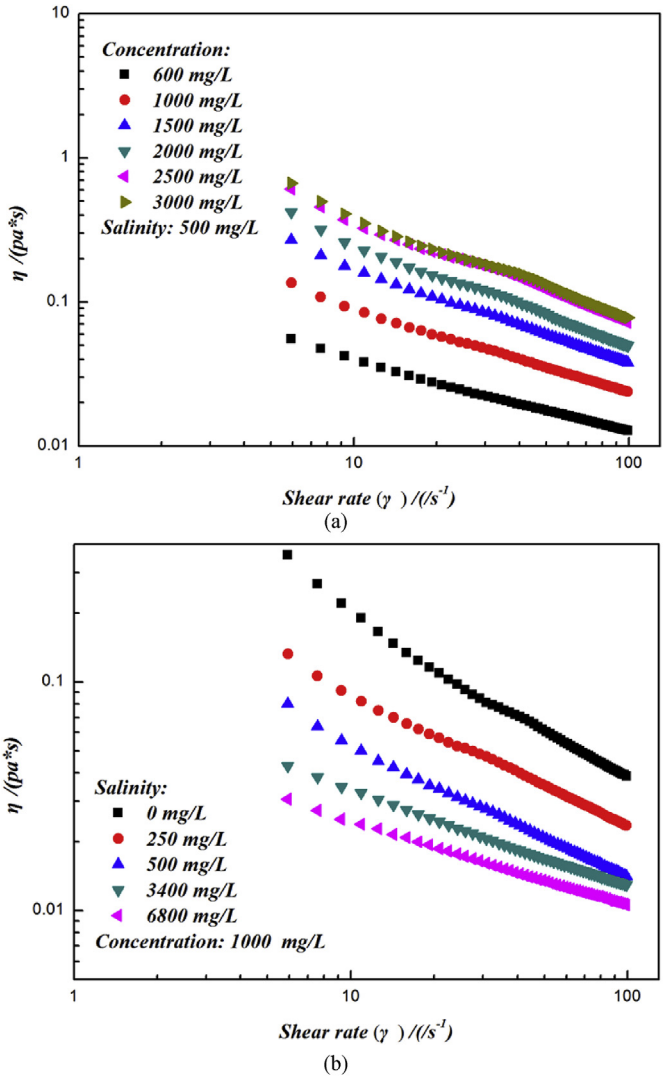


Fig. 5. Relationship curves between viscosity and shear rate of HPAM solution with different concentrations (a) and salinities (b).

monovalent salt can screen the charges on the polymer chains and the electrostatic repulsion effect in polymer chains would be weaker; thus the sizes of the polymer coil would be smaller, the crowded phenomenon would be weaker and the drop points shifted to lower angular frequencies.

To study the oscillatory shear behaviors clearly, the relaxation spectra should be plotted. The relaxation spectra can be calculated from the oscillatory shear spectra by using the relationship between storage modulus (***G′***), loss modulus (***G″***) and angular frequency (***ω***) given by the following integrals [28] (as shown in Eqs. (1) and (2)):

$$G'(\omega) = G_0 + \int_0^{\infty} H(\tau)\frac{\omega^2\tau^2}{1+\omega^2\tau^2}\left(\frac{d\tau}{\tau}\right) \quad (1)$$

$$G''(\omega) = \int_0^{\infty} H(\tau)\frac{\omega\tau}{(1+\omega^2\tau^2)}\left(\frac{d\tau}{\tau}\right) \quad (2)$$

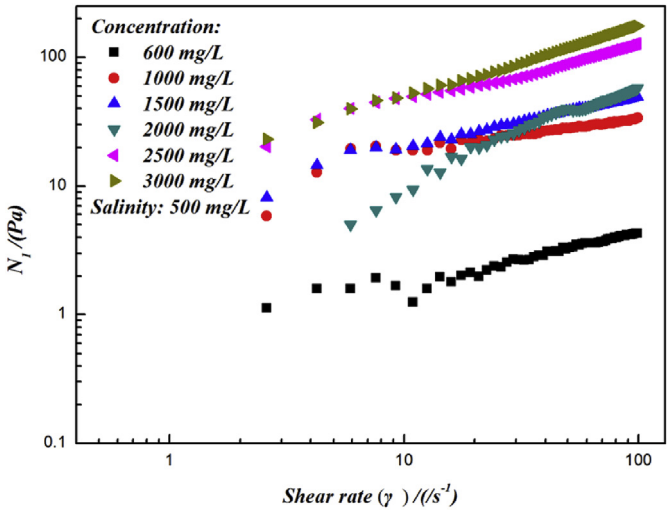
where ***H(τ)*** is the relaxation spectrum of distributions of the elements as a function of relaxation times, while G_0 is the equilibrium modulus which is zero for a viscoelastic liquid. Actually, this method of calculating the relaxation spectrum is an ill-posed problem and Kontogiorgos's numerical method was taken herein to avoid that problem [36,37]. After obtaining storage modulus data, the important work herein was calculation of the optimum regularization parameter. The regularization parameter relates to the filtering of the spectrum noise. In other words, it is a key step to maintaining balance between the

Table 1
Calculated power law parameters for HPAM solutions (R^2: Correlation coefficient).

Concentrations (mg/L)	Salinity (mg/L)	K^*10^2	n^*10^2	R^2*10^2
600	500	−85.31	47.11	99.08
1000	500	−38.20	37.00	98.93
1500	500	−0.12	27.95	98.86
2000	500	23.43	22.15	98.70
2500	500	40.51	21.94	98.41
3000	500	45.46	20.74	97.80
1000	0	19.73	17.11	97.62
1000	250	−38.90	37.32	98.86
1000	3400	−108.70	59.92	98.93
1000	6800	125.40	63.84	99.45

regularization error and the loss of resolution (smoothness) of the solution. The method that usually has been used is the L-curve method [34]. The solution norm ($\|x\|_2$) versus residual norms ($\|Ax-b\|_2$) for all valid regularization parameters were drawn. The vertical part of the curve corresponds to solutions that are sensitive to perturbation errors in measurements whereas the horizontal part of the curve corresponds to solutions where the regularization (calculation) error dominates. The optimum regularization parameter is located at the corner of the curve and this value is used to calculate the optimum least square solution of the spectrum. If the regularization parameter is smaller than the optimum point, an "arbitrary noise generator" would happen and would cause problems in the solution. However, if it is larger than the optimum point, some vital information from the experimental spectrum could be missed in the over-smoothed data [36]. Fig. 3 reproduces the results of the calculation of the optimum regularization parameter using the L-curve method for an HPAM solution with 1000 mg/L HPAM and 6800 mg/L NaCl, as an example.

The relaxation time spectra of HPAM solutions with different concentrations or different salinities were plotted in Fig. 4 a and b respectively, longer relaxation time corresponds to longer unit of segments, while a shorter relaxation time corresponds to a shorter unit of segments. The height of the curves in the relaxation time spectra indicates the number of units. In Fig. 4a, the number of segmental units clearly increased at first as the concentration increased. As the concentration was further increased, the peaks of segmental units shifted and split into several. One thing should be note that the multimodal structure of relaxation spectra may refer to polyelectrolyte properties of HPAM. The increase in the number of segmental units increase the elasticity of solution and the splitting multiple peaks of segmental units increase the elasticity-response in wide ranges. The molecular motion may easier happen at more frequencies than low concentrated one. For oil recovery, the injection of polymer flooding agents has been usually controlled by pressure; therefore the shear rate in different places may be different for the case of different flow channel. The increases of concentrations can make agents have high elasticity in the more place of underground. In Fig. 4b, the changes in relaxation behaviors can be classified as having two parts. At first, the number of segmental units increased as salinity increased from 0 to 500 mg/L, because



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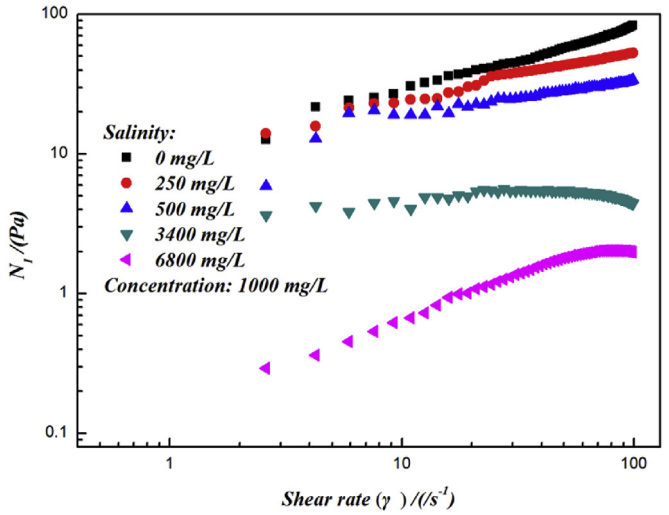

(b)

Fig. 6. Relationship between first normal stress difference measured by rheometer and shear rate of HPAM solution with different concentrations (a) and salinities (b).

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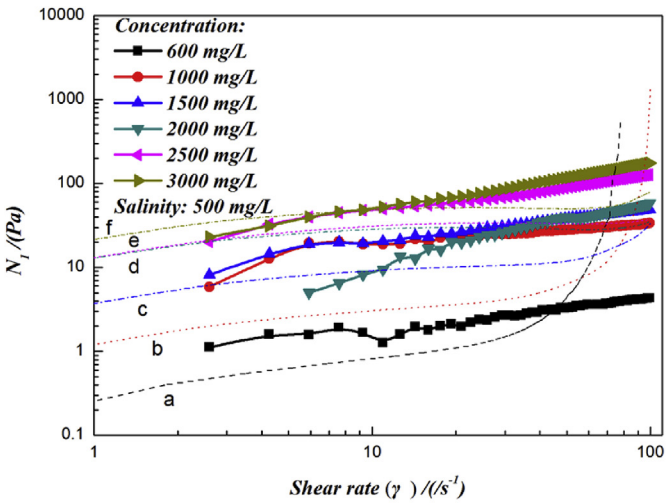
polymer chains would be flexible at low salinity. Then, the distribution of motion units became broader and the number of motion units in each angle frequency decreased as the salinity was further increased. This suggests that the elasticity-response of polymer flooding agents should be more average in underground but the elastic intensity decreases in some places.

3.2. Flow shear behaviors

In the flow shear test, the flow characterization can be determined through studying the relationship between shear viscosity and shear rate. As shown in Fig. 5, the shear viscosity of the HAPM solutions increased as the concentration increased, while it decreased as the salinity increased. The curves of shear viscosity versus shear rate could be well fitted by a well-known power law (as shown in Eq. (3)).

$$\eta(\gamma) = \mathbf{K}\gamma^{n-1} \tag{3}$$

where η is the shear viscosity, γ is the shear rate, $\boldsymbol{K}$ is the consistency index and $\boldsymbol{n}$ is the flow behavior index. The values of the parameters are shown in Table 1 where R is the correlation coefficient relative to fitting Eq. (3). According to Table 1, the flow behavior index decreased with increase of concentration, while it increased with increase of salinity. When flow behavior index is equal to 1, the fluid is Newton fluid. However, when flow behavior index is smaller than 1, the fluid is pseudoplastic fluid. The smaller flow behavior index is the stronger pseudoplasticity achieves. The pseudoplasticity of the HAPM solutions became stronger as the concentration increased; however, it was weaker as the salinity increased. The pseudoplasticity of the HAPM changed very fast when the concentrations were between 600 mg/L and 1500 mg/L. In dilute solution, the polymer chains cannot touch each other and pseudoplasticity mainly comes from orientation of the polymer chains under flow. In semi-dilute solution, the polymer chains can dynamically touch each other during flow [42], so the orientation of polymer chains, entanglement and disentanglement of polymers, both became the reasons that influenced the pseudoplasticity of the solutions. Furthermore, as the salinity was increased, the pseudoplasticity of the solutions became weaker. At a HPAM concentration of 1000 mg/L, entanglement could not happen, so the orientation of



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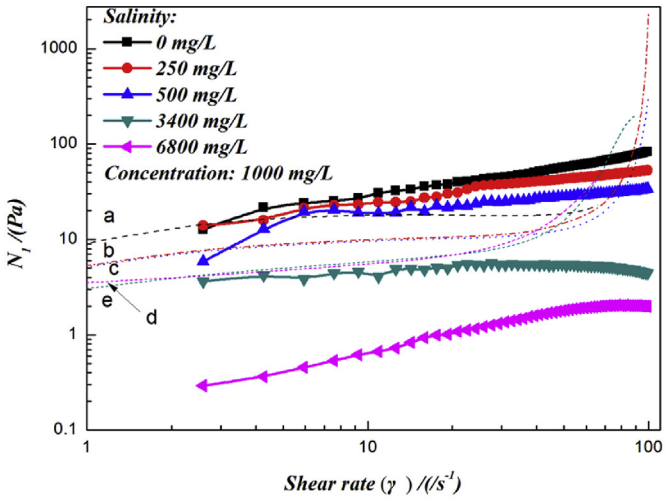

(b)

Fig. 7. Relationship curves between first normal stress difference calculated using Laun equation and shear rate of HPAM solution with different concentrations (a) and salinities (b).

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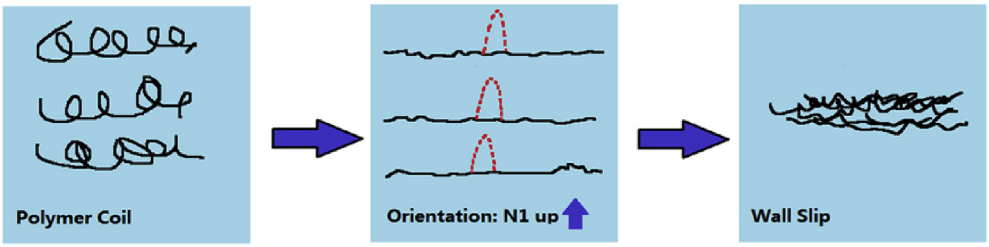

Fig. 8. Schematic diagram of wall slip phenomenon.

polymer chains was the primary reason that influence the pseudoplasticity of the solutions. When salinity was lower, the polymer chains are more rigid, so the orientation state of polymer chains would be maintained easier and pseudoplastic of solutions would be better.

The first normal stress difference has been thought to be an important parameter to characterize the elastic properties of polymer solutions. The first normal stress differences of the HPAM solutions with lower concentrations were measured in the rheometer (as shown in Fig. 6). The first normal stress differences increased as the concentration was increased and decreased as the salinity increased. As the concentration increases, the content of polymer chains in solution increases, so the elasticity increases and the first normal stress difference becomes larger. As the salinity increases, the molecular chains become more flexible, so their modulus would be smaller and the first normal stress differences would be smaller.

The Laun equation was usually used to calculate the first normal stress difference ($\mathbf{N_1}$) of polymer displacement agent [30] (as shown in Eq. (4)).

$$N_1 = 2G'\left[1+\left(\frac{G'}{G''}\right)^2\right]^{0.7}_{\omega=\gamma} \tag{4}$$

The plot of N_1 calculated using Laun equation vs. shear rate is plotted in Fig. 7. The N_1 calculated using Laun equation is fitted to trends of experimental data in low shear rate, but occurs deviation in high shear rate. However, Laun equation was obtained through studying melts, so this deviation may be caused by that the elasticity of dilute and semi-dilute polymer solutions is not as stronger as polymer melts. The Laun equation still can be used to estimate the first normal stress difference in low shear rate. Moreover, some literature reported that Laun equation was used to estimate the N1 of polymer oil displacement agents. However, most of polymer oil displacement agents are semi-diluted and diluted solutions. Thus, using Laun equation to estimate N_1 of polymer oil displacement agents is still limited, because polymer oil displacement agents flowing in porous medium are undergo high shear effect.

In high salinity (NaCl concentration: 3400 and 6800 mg/L), the N1 totally deviates Laun equation, because the experimental N1 decreased as the shear rates increased. This is caused by wall slip [43], which is that polymer solution changed from homogeneous to heterogeneous one, as shown in Fig. 8. At the beginning, the polymer chains become orientation. The recovery ability (red dot-line in the middle image of Fig. 8) increases so N1 increases. As shear rate further increases, the polymer chains aggregates into the central area of fluids, which makes homogeneous solution change to heterogeneous one (right image of Fig. 8). This result suggested that high salinity geology can reduce the elasticity of polymer flooding agents, which brings negative impact on polymer flooding.

4. Conclusions

The oscillatory shear tests showed that the storage and loss moduli increased as the HPAM concentration was increased and decreased as the salinity was increased. In addition, the tanδ was constant at low angular frequency, while it sharply dropped for angular frequencies exceeding a certain value. The drop points shifted to higher angular frequencies as the concentration was increased, while they shifted to lower angular frequencies as the salinity was increased. Calculating the relaxation spectra, we found that number of segmental units increase and peak of as concentration increases. Furthermore, as salinity increases, the quantity of motion units increases at first and then the distribution of motion units become broader. The flow shear test showed that the pseudoplasticity of the HAPM solutions became stronger as the concentration was increased, while it would be weaker as the salinity was increased. The N1 increased as the concentration was increased, but it decreased as the salinity was increased. When compare first normal stress difference measured by rheometer and N1 calculated using Laun equation, it was found that the Laun equation is well fitted to experimental result of the N1 measured by the rheometer when shear rate is lower, but deviation of Laun equation occured at higher shear rate. At the salinity of 3400 and 6800 mg/L, the N1 decreased at high shear rates, which are opposite to Laun equation's description.

Acknowledgments

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